REMARKS

Upon entry of the present amendment, claims 1-17 and 19-26 remain pending in the above-identified application and stand ready for further action on the merits.

Claim Amendments

Claims 1, 11 and 16 are amended, support for which resides at page 21, lines 23-25 of the specification. Claim 18 is canceled. Claim 20 is amended accordingly. No new matter is added by this amendment.

Approval of Drawings

Applicants note with appreciation the approval of the drawings in the application.

Rejection under 35 USC 103(a)

Claims 1-26 stand rejected under USC 103(a) as being unpatenteble over JP-8217972 in view of USP 5,109,052, USP 5,283,282, USP 5,470,902, USP 5,455,292, JP-11-181271, JP-9-169903, JP6-184398 and JP-5-706682.

In support of the rejection, the Examiner takes the position that "the rejection imposed in the previous Office action is maintained in its entirety." The Examiner also states that:

> "Asahi Chemical Co. Ltd. JP-8-217972 discloses in paragraph 0013 of the English translation (enclosed) that the preferred inherent viscosity range of 0.35-1.0. This range encompasses applicants' claimed inherent viscosity range of 0.37-0.41, which is equivalent to a reduced viscosity of 0.40-0.45. In the absence of a showing of criticality of the claimd reduced viscosity in the instant specification the rejection is not withdrawn."

This rejection is respectfully traversed.

In response, applicants note that an important characteristic feature of the present invention is, as the original claims recite, that the resin composition has a melt volume rate (hereinafter referred to as "MVR") of not less than 60 $cm^3/10$ min, which enables surprisingly desirable flow properties to be achieved.

The primary reference JP-8-217972 is totally silent regarding MVR, although it describes the values of melt flow rate (MFR) of the resin compositions at Tables 1 and 2. However, both MFR referenced in the reference, and MVR referenced in the present application, may be regarded as describing substantially the same property. The reason for that is shown below with reference to page 11 of ISO 1133 (entitled *Plastics – Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics* – Fourth edition 2005-06-01 - copy attached) in which the methods of calculation of MVR and MFR are described.

As explained in the ISO reference, MVR and MFR are indicative of flowability, both of which can be determined by using the same measurement device. The difference between MVR and MFR is only in that one (MVR) is determined by dividing the other (MFR) by a melt density of an object material. An alloy of polyamide/polyphenylene ether containing no filler, as disclosed in JP-8-217972 and the present application, has a melt density of approximately 1.0 g/cm^3. As a result, the MVR and MFR of such an alloy are substantially identical.

The values of MVR of resin compositions according to the present invention may be compared with the values of MFR of those according to JP-8-217972.

Specifically, the highest value of MFR described in JP-8-217972 is 28 g/10 min in Comparative Example 2 (see Table 1). The highest value of MFR in the working examples is just 21 g/10 min in Example 10. Therefore, it is evident that a resin composition of the reference is totally different from that of the present invention, which has a MVR of ***not less than*** 60 cm^3/10 min.

JP-8-217972 does not disclose a resin composition having both a large MFR and exhibiting excellent flowability as in the present invention. Furthermore, JP-8-217972 neither teaches nor suggests that a resin composition having highly desirable flowability is specifically suitable for a relay block as claimed.

Moreover, claim 1 of JP-8-217972 recites that a reduced viscosity of the polyamide of the molded article of the composition is 2.20 or more (in formic acid, 30°C). The Examiner is reminded that a larger reduced viscosity can result in a composition having lower flowability. It is clear from the description of the invention of the reference that the invention is preferably directed to a technical concept relating to a composition with *lower* flowability.

In contrast, the present invention is directed to a technical concept whereby a composition with *greater* flowability is preferred. Thus, JP-8-217972 teaches away from the present invention.

As discussed above, JP-8-217972 neither discloses nor suggests a resin composition having a MVR of *not less than* 60 cm^3/10 min. The reference thus teaches away from the present invention. In addition, the other cited references do not cure the above deficiency of the primary reference. Therefore, it would not be possible for a person skilled in the art to arrive at applicants' invention based on the teachings of the cited references.

In order to more clearly differentiate the present invention from the references, claims 1, 11 and 16 are amended so as to recite the limitation that the polyamide has a viscosity number of 100-130 ml/g (measured in 96% sulfuric acid in accordance with ISO307: 1994), which assists in achieving a desirable value of MVR of not less than 60 $cm^3/10$ min.

A resin composition suitable for a relay block and having the desired properties can be obtained by setting a viscosity number of a polyamide and a reduced viscosity of a polyphenylene ether in the resin composition to the specific ranges, and adjusting a MVR of the resin composition to not less than 60 $cm^3/10$ min. This fact is believed demonstrated by a comparison of the results of Example 1 with those of Comparative Example 1, and a comparison of the results of Example 3 with those of Example 4 (i.e., the example which has been changed to a comparative example by the previous amendment).

In addition, a comparison of the results of Example 3 with those of Comparative Example 2 reveals that the occurrence of melt wrinkles can be effectively suppressed by using a polyamide with a viscosity falling within the claimed range, even though they have similar values of MVR.

It would be extremely difficult for a person skilled in the art to obtain a resin composition having all of the features concerning viscosity number of the polyamide, a reduced viscosity of the polyphenylene ether, and the MVR of the resin composition recited in the present claims from the teachings of the references. Therefore, an excellent effect relating to highly desirable flowablity and suppression of melt wrinkles could not result from the teachings of the cited references in the absence of an improper hindsight analysis of the cited prior art.

The rejection is thus without basis and should be withdrawn.

The application is in condition for allowance, and an early indication of same earnestly is solicited.

Should there be any outstanding matters that need to be resolved in the present application, the Examiner is respectfully requested to contact John W. Bailey (Reg. No. 32,881) at the telephone number below, to conduct an interview in an effort to expedite prosecution in connection with the present application.

Payment in the amount of $110.00 is submitted herewith as payment for the requested one month extension of time.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies to charge payment or credit any overpayment to Deposit Account No. 02-2448 for any additional fees required under 37.C.F.R. §§1.16 or 1.14; particularly, extension of time fees.

Dated: April 16, 2007

Respectfully submitted,

By [signature] # 28 808 [illegible]

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Attachment: ISO 1133 (entitled *Plastics – Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics*(4th edition 2005-06-01)

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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet 1 of 1

Complete if Known	
Application Number	10/509,612-Conf. #3018
Filing Date	September 30, 2004
First Named Inventor	Takaaki MIYOSHI
Art Unit	1714
Examiner Name	P. A. Szekely
Attorney Docket Number	0152-0695PUS1

U.S. PATENT DOCUMENTS

Examiner Initials*	Cite No.[1]	Document Number: Number-Kind Code[2] (*if known*)	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear

FOREIGN PATENT DOCUMENTS

Examiner Initials*	Cite No.[1]	Foreign Patent Document: Country Code[3]-Number[4]-Kind Code[5] (*if known*)	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear	T[6]

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant. [1] Applicant's unique citation designation number (optional). [2] See Kinds Codes of USPTO Patent Documents at www.uspto.gov or MPEP 901.04. [3] Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). [4] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [5] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [6] Applicant is to place a check mark here if English language Translation is attached.

NON PATENT LITERATURE DOCUMENTS

Examiner Initials	Cite No.[1]	Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T[2]
	CA	ISO 1133, "Plastics-Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics (4th Edition 2005-06-01)	

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1]Applicant's unique citation designation number (optional). [2]Applicant is to place a check mark here if English language Translation is attached.

Examiner Signature		Date Considered	

Birch, Stewart, Kolasch & Birch, LLP

JWB/TK/enm

INTERNATIONAL STANDARD

ISO 1133

Fourth edition
2005-06-01

Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

Plastiques — Détermination de l'indice de fluidité à chaud des thermoplastiques, en masse (MFR) et en volume (MVR)

Reference number
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Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1133 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This fourth edition cancels and replaces the third edition (ISO 1133:1997), in which the clauses relating to temperature control have been revised. In addition, the clarity of the text has been improved.

Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

1 Scope

This International Standard specifies two procedures for the determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastic materials under specified conditions of temperature and load. Procedure A is a mass-measurement method. Procedure B is a displacement-measurement method. Normally, the test conditions for measurement of melt flow rate are specified in the material standard with a reference to this International Standard. The test conditions normally used for thermoplastics are listed in Annexes A and B.

The MVR will be found particularly useful when comparing materials of different filler content and when comparing filled with unfilled thermoplastics. The MFR can be determined from MVR measurements provided the melt density at the test temperature and pressure is known.

These methods are in principle also applicable to thermoplastics for which the rheological behaviour is affected during the measurement by phenomena such as hydrolysis, condensation or crosslinking, but only if the effect is limited in extent and only if the repeatability and reproducibility are within an acceptable range. For materials which show significantly affected rheological behaviour during testing, these methods are not appropriate. In such cases, the use of the viscosity number in dilute solution, determined in accordance with the relevant part of ISO 1628, is recommended for characterization purposes.

NOTE The rates of shear in these methods are much smaller than those used under normal conditions of processing, and therefore data obtained by these methods for various thermoplastics may not always correlate with their behaviour during processing. Both methods are used primarily in quality control.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1622-2, *Plastics — Polystyrene (PS) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 1628 (all parts), *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers*

ISO 1872-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 1873-2, *Plastics — Polypropylene (PP) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 2580-2, *Plastics — Acrylonitrile-butadiene-styrene (ABS) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 2897-2, *Plastics — Impact-resistant polystyrene (PS-I) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 4613-2, *Plastics — Ethylene/vinyl acetate (E/VAC) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 4894-2, *Plastics — Styrene/acrylonitrile (SAN) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 6402-2, *Plastics — Acrylonitrile-styrene-acrylate (ASA), acrylonitrile-(ethylene-propylene-diene)-styrene (AEPDS) and acrylonitrile-(chlorinated polyethylene)-styrene (ACS) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 7391-2, *Plastics — Polycarbonate (PC) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 8257-2, *Plastics — Poly(methyl methacrylate) (PMMA) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 8986-2, *Plastics — Polybutene (PB) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 9988-2, *Plastics — Polyoxymethylene (POM) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 10366-2, *Plastics — Methyl methacrylate-acrylonitrile-butadiene-styrene (MABS) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

ISO 15494, *Plastic piping systems for industrial applications — Polybutene (PB), polyethylene (PE) and polypropylene (PP) — Specifications for components and the system — Metric series*

ISO 15876-3, *Plastics piping systems for hot and cold water installations — Polybutylene (PB) — Part 3: Fittings*

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
melt mass-flow rate
MFR
rate of extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of temperature, load and piston position in the barrel of an extrusion plastometer, the rate being determined as the mass extruded over a specified time

NOTE The correct SI units are decigrams per minute (dg/min). However, grams per 10 minutes (g/10 min) have customarily been used in the past and are also acceptable.

3.2
melt volume-flow rate
MVR
rate of extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of temperature, load and piston position in the barrel of an extrusion plastometer, the rate being determined as the volume extruded over a specified time

NOTE The correct SI units are cubic decimetres per minute (dm^3/min). More commonly used units, which are also acceptable, are cubic centimetres per 10 minutes (cm^3/10 min).

3.3
load
combined mass of piston and added weight, as specified by the conditions of the test

NOTE It is expressed in kilograms (kg).

4 Principle

The melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) are determined by extruding molten material from the barrel of a plastometer under preset conditions of temperature and load. For melt mass-flow rate, timed segments of the extrudate are weighed and the extrudate rate is calculated in g/10 min and recorded. For melt volume-flow rate, the distance that the piston moves in a specified time or the time required for the piston to move a specified distance is measured to generate data in cm^3/10 min. Melt volume-flow rate may be converted to melt mass-flow rate, or *vice-versa*, if the density of the material is known under the conditions of the test.

5 Apparatus

5.1 Extrusion plastometer

The basic apparatus comprises an extrusion plastometer operating at a fixed temperature. The general design is as shown in Figure 1. The thermoplastic material, which is contained in a vertical cylinder, is extruded through a die by a piston loaded with a known weight. The apparatus consists of the following essential parts.

5.1.1 Cylinder, fixed in a vertical position (see 5.1.5). The cylinder shall be manufactured from a material resistant to wear and corrosion up to the maximum temperature of the heating system, and the finish, properties and dimensions of its surface shall not be affected by the material being tested. For particular materials, measurements may be required at temperatures up to 450 °C. The cylinder shall have a length between 115 mm and 180 mm and an internal diameter of 9,550 mm ± 0,025 mm. The base of the cylinder shall be thermally insulated in such a way that the area of exposed metal is less than 4 cm^2, and it is recommended that an insulating material such as Al_2O_3, ceramic fibre or another suitable material be used in order to avoid sticking of the extrudate.

The bore shall be hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1) and shall be manufactured by a technique that produces a surface roughness of less than Ra (arithmetical mean deviation) = 0,25 µm (see ISO 4287). If necessary, a piston guide shall be provided to keep friction caused by misalignment of the piston down to a minimum.

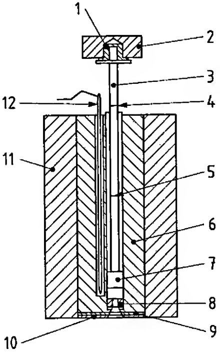
NOTE Excessive wear of the cylinder, piston head, and piston is an indication of misalignment of the piston. Regular checking for wear and change to the surface appearance of the cylinder, piston and piston head is required to ensure these items are within specification.

5.1.2 Piston, having a working length at least as long as the cylinder. The piston shall be manufactured from a material resistant to wear and corrosion up to the maximum temperature of the heating system and its properties and dimensions shall not be affected by the material being tested. The piston shall have a head 6,35 mm ± 0,10 mm in length. The diameter of the head shall be less than the internal diameter of the cylinder by 0,075 mm ± 0,010 mm. The upper edge shall have its sharp edge removed. Above the head, the piston

shall be relieved to ⩽ 9 mm diameter. A stud may be added at the top of the piston to support a removable weight, but the piston shall be thermally insulated from the weight. Along the piston stem, two thin annular reference marks shall be scribed 30 mm apart and so positioned that the upper one is aligned with the top of the cylinder when the distance between the lower edge of the piston head and the top of the die is 20 mm. These annular marks on the piston are used as reference points during the measurements (see 8.4 and 9.5).

To ensure satisfactory operation of the apparatus, the cylinder and the piston head shall be made of materials of different hardness. It is convenient for ease of maintenance and renewal to make the cylinder of the harder material.

The piston may be either hollow or solid. In tests with very low loads, the piston may need to be hollow, otherwise it may not be possible to obtain the lowest prescribed load. When the test is performed with the higher loads, a solid piston or hollow piston with guides shall be used.



Key

1 insulation
2 removable weight
3 piston
4 upper reference mark
5 lower reference mark
6 cylinder
7 piston head
8 die
9 die retaining plate
10 insulating plate
11 insulation
12 temperature sensor

Figure 1 — Typical apparatus for determining melt flow rate, showing one possible configuration

5.1.3 Temperature-control system: For all cylinder temperatures that can be set, the temperature control shall be such that, between 10 mm above the top of the die and 75 mm above the top of the die, the temperature differences measured do not exceed those given in Table 1 throughout the duration of the test.

NOTE The temperature may be measured with thermocouples, platinum-resistance sensors, or mercury-in-glass thermometers embedded in the wall. If the apparatus is equipped in this way, the temperature may not be exactly the same as that in the melt, but the temperature-control system may be calibrated (see 7.1) to read in melt temperature.

The temperature-control system shall allow the test temperature to be set in steps of 0,2 °C or less.

Table 1 — Maximum allowable variation in temperature with distance and with time throughout the test

Test temperature, T °C	Maximum variation in test temperature [a]	
	with distance at between 10 mm and 75 mm above the die surface °C	with time at 10 mm above the die surface [b] °C
$125 \leqslant T < 250$	± 2,0	± 0,5 [c]
$250 \leqslant T < 300$	± 2,5	± 0,5
$300 \leqslant T$	± 3,0	± 1,0

[a] Variation is over the normal time of a test, typically less than 25 min, and can be verified during calibration of the equipment.

[b] When using a 4 mm length die (see 5.1.4), the readings should be made 14 mm above the die surface.

[c] A value of 0,2 °C is preferred since it gives better reproducibility. It is intended that the value of 0,2 °C will become a requirement at the next revision of this International Standard.

5.1.4 Die, made of tungsten carbide or hardened steel, 8,000 mm ± 0,025 mm in length. The interior of the bore shall be manufactured circular, straight and uniform in diameter such that in all positions it is within ± 0,005 mm of a true cylinder of nominal diameter 2,095 mm. The bore diameter shall be checked regularly with a go/no-go gauge. If outside the tolerance limits, the die shall be discarded. The die shall have ends that are flat, perpendicular to the axis of the bore and free from visible machining marks.

If testing materials with a melt mass-flow rate > 75 g/10 min or a melt volume-flow rate > 75 cm^3/10 min, a half-height, half-diameter die 4,000 mm ± 0,025 mm in length and with a bore of nominal diameter 1,050 mm ± 0,005 mm should preferably be used. No spacer shall be used with this die to increase the apparent length to 8,00 mm.

For testing potentially corrosive materials, dies made of cobalt-chromium-tungsten alloy, chromalloy, synthetic sapphire or other suitable materials may be used.

The bore shall be hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1) and shall be manufactured by a technique that produces a surface roughness of less than Ra (arithmetical mean deviation) = 0,25 µm (see ISO 4287). The die shall not project beyond the base of the cylinder (see Figure 1) and shall be mounted so that its bore is co-axial with the cylinder bore.

The flat surfaces of the die shall be checked to ensure that the area around the bore is not chipped. Any chipping will cause errors and chipped dies shall be discarded.

5.1.5 Means of setting and maintaining the cylinder truly vertical: A two-directional bubble level, set normal to the cylinder axis, and adjustable supports for the apparatus are suitable for the purpose.

NOTE This is to avoid excessive friction caused by the piston leaning to one side or bending under heavy loads. A dummy piston with a spirit level on its upper end is also a suitable means of checking conformity with this requirement.

5.1.6 Load: A set of removable weights, which may be adjusted so that the combined mass of the weights and the piston gives the selected nominal load to an accuracy of ± 0,5 %, are mounted on top of the piston.

Alternatively, a mechanical loading device combined with, for example, a load cell, providing the same level of accuracy as the removable weights, may be used.

5.2 Accessory equipment

5.2.1 General

5.2.1.1 Packing rod, made of non-abrasive material, for introducing test samples into the cylinder.

5.2.1.2 Cleaning equipment (see 7.2).

5.2.1.3 Go/no-go gauge, one end having a pin with a diameter equal to that of the die bore minus the allowed tolerance (go gauge) and the opposite end having a pin with a diameter equal to that of the die bore plus the allowed tolerance (no-go gauge). The pin gauge shall be sufficiently long to check the full length of the die using the go gauge.

5.2.1.4 Temperature-calibration device (mercury-in-glass thermometer, thermocouple, platinum-resistance sensor or other temperature-measuring device). The temperature sensor shall have a temperature readout resolution of 0,1 °C or better. Calibrate the temperature-indicating device using for example a light-gauge probe-type thermocouple or a platinum-resistance sensor having a short sensing length. The thermocouple should be encased in a metallic sheath having a diameter of approximately 1,6 mm with its hot junction grounded to the end of the sheath.

5.2.1.5 Die plug: A device shaped at one end so that it effectively blocks the die exit and prevents drool of molten material while allowing rapid removal prior to initiation of the test, e.g. a plug attached to a compressed spring.

5.2.1.6 Piston/weight support, of sufficient length to hold the piston so that the lower reference mark is 25 mm above the top of the cylinder.

5.2.2 Equipment for procedure A (see Clause 8)

5.2.2.1 Cutting tool, for cutting off extruded sample. A sharp-edged spatula has been found suitable.

5.2.2.2 Timer, accurate to ± 0,1 s for melt mass-flow rates ⩽ 100 g/10 min and to ± 0,01 s for melt mass-flow rates > 100 g/10 min. Compare with a calibrated timing device over a period of 15 min to 20 min and ensure that the tolerance is within ± 0,07 % of the total time measured.

NOTE Procedure A is not recommended for measurement of melt mass-flow rates > 100 g/10 min unless using a half-height, half-diameter die.

5.2.2.3 Balance, accurate to ± 0,5 mg.

5.2.3 Equipment for procedure B (see Clause 9)

5.2.3.1 Measurement equipment, for the automatic measurement of distance and time for the piston movement, using single or multiple determinations for a single charge (see Table 2).

Table 2 — Piston distance and time measurement accuracy requirements

MFR (g/10 min) or **MVR** (cm^3/10 min)[a]	**Distance** (mm)	**Time** (s)
0,1 to 1,0	± 0,02	± 0,1
> 1,0 to 100	± 0,1	± 0,1
> 100	± 0,1	± 0,01

[a] For multiple measurements in a single charge regardless of the MFR or MVR of the material, the requirements are the same as for MFR or MVR > 100.

6 Test sample

6.1 Sample form

The test sample may be in any form that can be introduced into the bore of the cylinder, for example granules, strips of film, powder or sections of moulded or extruded parts. The test sample may fill the cylinder bore to a height of 75 mm prior to starting the test.

NOTE 1 In order to ensure void-free extrudates when testing powders, it may be necessary to first compress the material to a preform or pellets.

NOTE 2 The form of the test sample can be a significant factor in determining the reproducibility. The form of the test sample should therefore be controlled to improve the comparability of inter-laboratory results and to reduce the variability between runs.

6.2 Conditioning

The test sample shall be conditioned and, if considered necessary, stabilized prior to testing, in accordance with the appropriate material specification standard.

7 Temperature-calibration, cleaning and maintenance of the apparatus

7.1 Calibration of the temperature-control system

7.1.1 Calibration procedure

It is necessary to verify regularly the accuracy of the temperature-control system (5.1.3).

Set the temperature-control system to the required temperature (as indicated by the control temperature-indicating device). Charge the cylinder with a quantity of the material to be tested, or a material representative thereof (see 7.1.2), using the same technique as for a test (see 8.3). Five minutes after completing the charging of the material, introduce the calibrated temperature-indicating device (5.2.1.4) into the sample chamber and immerse it in the material therein until the sensor is 10 mm from the upper face of the die. After a further interval of not less than 4 min and not more than 10 min, correct the temperature indicated by the control temperature-indicating device by algebraic addition of the difference between the temperatures read on the two temperature sensors. It is also necessary to verify the temperature profile along the cylinder. For this, measure the temperature of the material also at 30 mm, 50 mm and 75 mm above the upper face of the die. Check the temperature over time as well as distance for conformance to Table 1. If using a calibration thermometer as the temperature-indicating device, preheat the thermometer to the same temperature as that being measured.

NOTE It is recommended that, in verifying the temperature profile along the cylinder, the measurements are started at the highest point above the die.

An alternative technique for calibration is to use a sheathed thermocouple or platinum-resistance temperature sensor with tip diameter of 9,4 mm ± 0,1 mm for insertion in the bore without material present. Another technique is use of a piston provided with thermocouples at heights of 10 mm, 30 mm, 50 mm and 75 mm above the die, which can be inserted completely in the bore and fits the bore closely. This configuration will allow temperature calibration of the apparatus and verification of the temperature profile at the same time.

7.1.2 Calibration material

It is essential that the material used during calibration be sufficiently fluid to permit, for instance, a mercury-filled thermometer bulb to be introduced without excessive force or risk of damage. A stable material with an MFR of greater than 45 g/10 min (2,16 kg load) at the calibration temperature has been found suitable.

If such a material is used for calibration purposes in place of a more viscous material which is to be tested, the dummy material shall have a thermal diffusivity similar to that of the material to be tested, so that warm-up behaviour is similar. It is necessary that the quantity charged for calibration be such that, when the calibration temperature sensor is subsequently introduced, the appropriate length of the sensor stem is immersed for accurate temperature measurement. This can be checked by inspecting the upper edge of the material coating the end of the calibration temperature sensor, removing the sensor from the cylinder if necessary.

7.2 Cleaning the apparatus

WARNING — The operating conditions may entail partial decomposition of the material under test or any material used to clean the instrument, or cause them to release dangerous volatile substances, as well as presenting the risk of burns. The user of this International Standard is therefore responsible for keeping him- or herself informed of possible risks of accident and for providing appropriate means of protection.

The apparatus shall be cleaned thoroughly after each determination. The cylinder may be cleaned with cloth patches. The piston shall be cleaned while hot with a cotton cloth. The die may be cleaned with a closely fitting brass reamer, high-speed drill bit of 2,08 mm diameter, or wooden peg. Pyrolytic cleaning of the die in a nitrogen atmosphere at about 550 °C may also be used. Abrasives or materials likely to damage the surface of the piston, cylinder or die shall not be used. Take care that the cleaning procedure used does not affect the cylinder and die dimensions or surface finish. The die bore shall be checked with a go/no-go gauge after cleaning. When testing polyolefins, do not use copper-containing materials, e.g. brass brushes, to clean the cylinder, piston or die as this may accelerate degradation of the polymer.

If solvents are used to clean the cylinder, take care that any effect they may have on the next determination is negligible.

8 Procedure A: mass-measurement method

8.1 Selection of temperature and load

Refer to Annexes A and B. Use the conditions in Table B.1 if the material is listed there. The material standard takes precedence over this International Standard. If no material standard exists, use an appropriate set of conditions from Table A.1 based on knowledge of the melting point of the material or the processing conditions recommended by manufacturer.

8.2 Cleaning

Clean the apparatus (see 7.2). Before beginning a series of tests, ensure that the cylinder and piston have been at the selected temperature for not less than 15 min.

8.3 Selection of sample mass and charging cylinder

Charge the cylinder with 3 g to 8 g of the sample according to the anticipated MFR or MVR (see Table 3). During the charging, compress the material with the packing rod (5.2.1.1), using hand pressure. For materials susceptible to oxidative degradation, ensure the charge is as free from air as possible. Complete the charging process in < 1 min. The preheat time of 5 min begins after charging of the cylinder has been completed.

NOTE Variations in the packing pressure used to compress the material in the cylinder can cause poor repeatability of results. Use of the same mass of sample for the analysis of materials of similar MFR or MVR will reduce variability in the data.

Immediately put the piston in the cylinder. The piston may be either unloaded or preloaded with the test weight or, for materials with high flow rates, a smaller weight. If the melt mass-flow rate or melt-volume flow rate of the material is high, that is, more than 10 g/10 min or 10 cm^3/10 min, the loss of sample during preheating will be appreciable. In this case, use an unloaded piston or one carrying a smaller load during the preheating period, and then change to the desired load at the end of the 5 min preheating time. In the case of very high

melt flow rates, a weight support should preferably be used and a die plug may be necessary. If the die plug is used and less than the desired load is on the piston, add the desired load and allow the material to stabilize for a few seconds before removing the die plug. If a weight support and die plug are both used, remove the weight support first.

NOTE To minimize the risk of burns from hot material coming out of the die rapidly, it is recommended that heat-resistant gloves be worn during the removal of the die plug.

Table 3 — Guidelines for experimental parameters

MFR (g/10min) or **MVR** (cm^3/10min) [a]	**Mass of test sample in cylinder** [b, c] g	**Extrudate cut-off time-interval** s
$\geqslant$ 0,1 but $\leqslant$ 0,5	3 to 5	240
> 0,5 but $\leqslant$ 1	4 to 6	120
> 1 but $\leqslant$ 3,5	4 to 6	60
> 3,5 but $\leqslant$ 10	4 to 8	30
> 10	4 to 8	5 to 15 [d]

[a] It is recommended that a melt flow rate should not be measured if the value obtained in this test is less than 0,1 g/10 min (MFR) or 0,1 cm^3/10 min (MVR). Melt mass-flow rates greater than 100 g/10 min should only be measured if the timer resolution is 0,01 s and procedure B is used when a standard 8,00 mm die is used. Alternatively, the half-height, half-diameter die may be used with procedure A (see 5.1.4).

[b] When the density of the material is greater than 1,0 g/cm^3, it may be necessary to increase the mass of the test sample. Use the low mass values for low-density materials.

[c] Sample mass is a significant factor in determining the repeatability of this test and may need to be controlled to 0,1 g to reduce variability between runs.

[d] To achieve adequate repeatability when testing materials having an MFR greater than 25 g/10 min (or MVR greater than 25 cm^3/10 min), it may be necessary either to control and measure cut-off intervals to an accuracy of less than 0,1 s or to use procedure B.

8.4 Measurements

8.4.1 Five minutes after completing the introduction of the test sample, place the selected weight on the piston, if it was unloaded or under-loaded. During this time, check that the temperature has returned to that selected. Allow the piston to descend under gravity until a bubble-free filament is extruded; this may be done before or after loading, depending on the actual viscosity of the material. It is strongly recommended that forced purging of the sample, done either manually or by using extra weights, before commencement of the test be avoided. If any forced purging is required (i.e. to complete the procedure within the specified time limit), it shall be finished at least 2 min before the start of the test. Any purging shall be carried out within a period of 1 min. Cut off the extrudate with the cutting tool (5.2.2.1), and discard. Continue to allow the loaded piston to descend under gravity. When the lower reference mark has reached the top edge of the cylinder, start the timer (5.2.2.2), and simultaneously cut off the extrudate with the cutting tool and again discard.

NOTE For some materials, shorter times may be required to prevent degradation and for high melting point, high T_g, low thermal conductivity and similar materials, e.g. PMMA, longer times may be needed to obtain repeatable results.

8.4.2 Collect successive cut-offs in order to measure the extrusion rate at a given time-interval. Depending on the melt mass-flow rate, choose a time-interval so that the length of a single cut-off is not less than 10 mm and preferably between 10 mm and 20 mm (see cut-off time-intervals in Table 3 as a guide).

For low values of MFR (and MVR) and/or materials which exhibit a relatively high degree of die swell, it may not be possible to take a cut-off with a length of 10 mm or more within the maximum time-interval of 240 s. In such cases, procedure A may be used, but only if the mass of each cut-off obtained in 240 s is greater than 0,04 g. If not, procedure B shall be used.

8.4.3 Stop cutting when the upper mark on the piston stem reaches the top edge of the cylinder. Discard any cut-off containing visible air bubbles. After cooling, weigh individually, to the nearest 1 mg, the remaining cut-offs, preferably three or more, and calculate their average mass. If the difference between the maximum and the minimum values of the individual weighings exceeds 15 % of the average, discard the results and repeat the test on a fresh portion of the sample. For materials suspected to be non-homogeneous, such as recycled material, it is recommended that the cut-offs be weighed in order of extrusion. If a continuous change in mass is observed, this shall be reported as unusual behaviour (see Clause 12).

8.4.4 The time between the end of charging the cylinder and the last measurement shall not exceed 25 min for any material. For some materials, this time may need to be reduced to prevent degradation or crosslinking of the material during the test.

8.5 Expression of results

8.5.1 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}\left(T, m_{\mathrm{nom}}\right) = \frac{600m}{t}$$

where

T is the test temperature, in degrees Celsius;

m_{nom} is the nominal load, in kilograms;

m is the average mass, in grams, of the cut-offs;

t is the cut-off time-interval, in seconds;

600 is the factor used to convert grams per second into grams per 10 min (600 s).

8.5.2 The melt volume-flow rate may also be calculated from the melt mass-flow rate using the following equation:

MVR = MFR/Melt density of material

8.5.3 Express the result to two significant figures (three significant figures for results < 10,0) and record the test temperature and load used (e.g. 190/2,16).

9 Procedure B: displacement-measurement method

9.1 Selection of temperature and load

See 8.1.

9.2 Minimum piston displacement distance

For improved accuracy and repeatability of measurements of low melt flow rate materials, e.g. MFR < 1,0 g/10 min or MVR < 1,0 cm^3/10 min, the following minimum piston displacement distances are suggested:

MFR or MVR range	Distance (minimum)
> 0,10 but ⩽ 0,15	3 mm
> 0,15 but ⩽ 0,40	4 mm
> 0,40 but ⩽ 1,0	10 mm
> 1,0	20 mm

9.3 Timer

Where the displacement measurement and/or timing device used for piston displacement measurement makes physical contact with the piston or weight, the load shall not be altered by more than 0,5 % of the total load.

9.4 Preparation for the test

As in procedure A (follow 8.2 to 8.4.1).

9.5 Measurements

9.5.1 Do not start taking measurements before the lower reference mark has reached the top edge of the cylinder.

9.5.2 Take measurements as follows:

a) Either measure the distance moved by the piston at predetermined times.

NOTE For some materials, results can vary depending on the distance moved by the piston. For improved repeatability, it is critical to maintain the same distance moved between individual runs.

b) Or measure the times taken by the reference mark to cover a specified distance.

Stop the measurements when the upper mark on the piston stem reaches the edge of the cylinder.

9.5.3 The time between the end of charging the cylinder and the last measurement shall not exceed 25 min for any material. For some materials, this time may need to be reduced to prevent degradation or crosslinking of the material during the test.

9.6 Expression of results

9.6.1 The melt volume-flow rate (MVR), expressed in cubic centimetres per 10 min, is given by the equation

$$\mathrm{MVR}(T, m_{\mathrm{nom}}) = \frac{A\,600\,l}{t}$$

where

T is the test temperature, in degrees Celsius;

m_{nom} is the nominal load, in kilograms;

A is the mean of the cross-sectional areas of the cylinder and the piston head, in square centimetres (nominally 0,711 cm^2 but, based on the tolerances allowed on the cylinder diameter, the product $A \times 600$ can vary from 424 to 428 and shall be calculated for the geometry of the cylinder actually used for the test);

t is the predetermined time of measurement or the mean value of the individual time measurements, in seconds (see 9.5.2);

l is the predetermined distance moved by the piston or the mean value of the individual distance measurements, in centimetres (see 9.5.2).

9.6.2 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}(T, m_{\mathrm{nom}}) = \frac{A\,600\,l\,\rho}{t}$$

where

T, m_{nom}, A, t and l are as defined in 9.6.1;

ρ is the density, in grams per cubic centimetre, of the melt at the test temperature and is given by the equation

$$\rho = \frac{m}{Al}$$

m being the mass, in grams, determined by weighing, of extrudate expelled by a piston movement of l cm.

9.6.3 Express the result to two significant figures (three significant figures for results < 10,0) and record the test temperature and load used (e.g. 190/2,16).

10 Flow rate ratio (FRR)

The ratio of two values of MFR (or MVR) obtained for a material tested at the same temperature but with different loads is called the flow rate ratio, e.g.

$$\mathrm{FRR} = \frac{\mathrm{MFR}(190/10{,}0)}{\mathrm{MFR}(190/2{,}16)}$$

It is commonly used as an indication of the way in which the rheological behaviour of a thermoplastic is influenced by the molecular mass distribution of the material.

NOTE The conditions to be used for the determination of the flow rate ratio are given in the appropriate material standards.

Express results to two significant figures (or three if both the MFR and MVR are expressed to three).

11 Precision

When the method is used with certain materials, consideration shall be given to the factors which may influence the magnitude of the measured values and may lead to a decrease in repeatability. Such factors include the following:

a) thermal degradation or crosslinking of the material, causing the melt flow rate to change during the preheating or test period (powdered materials requiring long preheating times are sensitive to this effect and, in certain cases, the inclusion of stabilizers is necessary to reduce the variability);

b) with filled or reinforced materials, the distribution or orientation of the filler may affect the melt flow rate.

The precision of the method is not known because interlaboratory data are not available. A single precision statement would not be suitable because of the number of materials covered. However, a coefficient of variation of about ± 10 % could be expected between laboratories and ± 5 % within a laboratory.

12 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the test sample, including the physical form of the material with which the cylinder was charged;

c) the details of conditioning;

d) the details of any stabilization (see 6.2);

e) the die dimensions;

f) the temperature and load used in the test;

g) the pre-heat time used;

h) for procedure A, the masses of the cut-offs and the cut-off time-intervals or, for procedure B, the predetermined time of measurement or distance moved by the piston and the corresponding measured values of the distance moved by the piston or the time of measurement;

i) the melt mass-flow rate, in grams per 10 min, or the melt volume-flow rate, in cubic centimetres per 10 min, expressed to two significant figures (three significant figures for results < 10,0) (when more than one value has been obtained, all the individual values shall be reported, as well as the mean value);

j) if appropriate, the flow rate ratio (FRR);

k) a report of any unusual behaviour of the test sample, such as discoloration, sticking, extrudate distortion or unexpected variation in melt flow rate;

l) the date of the test.

Annex A
(normative)

Test conditions for MFR and MVR determinations

The conditions used shall be as indicated in the appropriate material specification standard. Table A.1 indicates test temperatures and loads that have been found useful.

Table A.1

Test temperature, T, °C
100
125
150
190
200
220
230
235
240
250
260
265
275
280
300

Nominal load (combined), m_{nom}, kg
0,325
1,20
2,16
3,80
5,00
10,00
21,60

NOTE 1 It is recommended that temperatures and loads from this table be used for new thermoplastic materials. Any combination of temperature and load may be used. However, the choice of temperature and load combination(s) should be based on the rheological properties of the material and be defined in the material specification standard.

NOTE 2 The code-letters that were used in the past to describe combinations of temperature and load have been eliminated from this table and will be phased out of Table B.1.

Annex B
(informative)

Conditions specified in International Standards for the determination of the melt flow rate of thermoplastic materials

Table B.1 indicates test conditions that are currently specified in relevant International Standards or by agreement with the relevant ISO committee. All standards are subject to revision, and the test conditions should be verified with the current material standard prior to testing. Other test conditions not listed here may be used, if necessary, for a particular material (see Annex A).

Table B.1

International Standard (see Clause 2)	Materials	Conditions (code-letter)	Test temperature, T °C	Nominal load (combined), m_{nom} kg
ISO 1622-2	PS	H	200	5,00
ISO 1872-2	PE [a]	D	190	2,16
ISO 1872-2	PE [a]	E	190	0,325
ISO 1872-2	PE [a]	G	190	21,60
ISO 1872-2	PE [a]	T	190	5,00
ISO 1873-2	PP [a]	M	230	2,16
ISO 1873-2	PP [a]		230	5,00
ISO 2580-2	ABS	U	220	10,00
ISO 2580-2	ABS		240	10,00
ISO 2580-2	ABS		265	10,00
ISO 2897-2	PS-I	H	200	5,00
ISO 4613-2	EVAC [b]	B	150	2,16
ISO 4613-2	EVAC [b]	D	190	2,16
ISO 4613-2	EVAC [b]	Z	125	0,325
ISO 4894-2	SAN	U	220	10,00
ISO 6402-2	ASA, ACS, AEDPS	U	220	10,00

Table B.1 (*continued*)

International Standard (see clause 2)	Materials	Conditions (code letter)	Test temperature, T °C	Nominal load (combined), m_{nom} kg
ISO 6402-2	ASA, AEDPS		240	10,00
ISO 6402-2	ASA, AEDPS		265	10,00
ISO 7391-2	PC [c]	W	300	1,20
ISO 8257-2	PMMA	N	230	3,80
ISO 8986-2	PB	D	190	2,16
ISO 8986-2	PB	F	190	10,00
ISO 15876-3	PB	T	190	5,00
ISO 9988-2	POM	D	190	2,16
ISO 15494	PP	T	190	5,00
ISO 10366-2	MABS	U	220	10,00

[a] Melt density values for this material are included in the material standard.

[b] For EVAC, set of conditions B or D is used for MFRs up to 100 g/10 min; set of conditions Z is used for MFRs > 100 g/10 min under set of conditions B.

[c] Sample must be dried to ⩽ 0,02 % moisture level prior to testing.